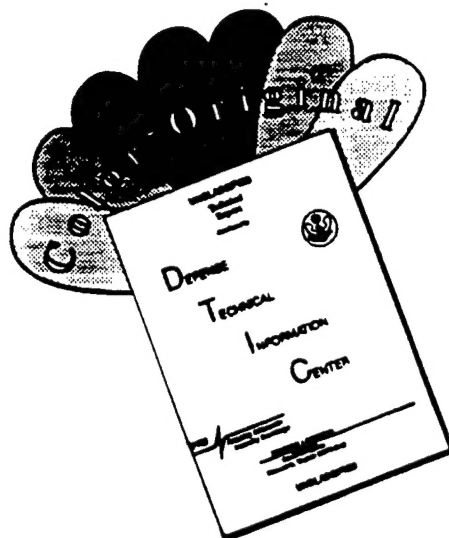


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Next Generation Ion Beam Analysis

Final Technical Report

Dr. Peter Revesz and Prof. Michael O. Thompson

February 7, 1996

U.S. Army Research Office

**P-33751-MS-RIP
DAAH04-95-1-0019**

Cornell University

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1 Foreword

Light element analysis, particularly hydrogen (H) and deuterium (D) but generally for any $Z \leq 14$, is a critical need for a large class of research and development projects. Most analytical techniques, including for example MeV ion beams and X-ray fluorescence, have difficulties with low Z elements due to relatively small interaction cross-sections. While H and D can be observed in the forward scattering geometry with ion scattering, two severe drawbacks remain: low depth resolution resulting from the need for stopper foils and relatively small depths which can be analyzed.

In order to mitigate these drawbacks and to advance our existing ion beam based analysis tools, we have completed the design, building and testing phases for a new tool – a variable geometry time-of-flight energy telescope and scattering chamber. In parallel, we have developed the software tools required to analyze, interpret, and model the spectra collected by this new instrument.

The instrument presently has the following features:

1. Custom designed scattering chamber:
 - variable (0-180°) detector geometry
 - variable (0-360°) sample tilt with tilt sensor
 - fast sample change cycle (~ 10 minutes)
2. ToF detector:
 - custom designed solid state detector and backup pump attachments
 - detection solid angle of 0.31 mSr
 - flight length of 980 mm
3. Electronics:
 - detector energy resolution of ~ 20 keV
 - ToF timing resolution of < 1 ns
4. ToF utility software:
 - fast visualization of data
 - state-of-the-art data compression
 - 2D spectrum manipulation for easy data reduction

2 Statement of the Problem Studied

The most widely used MeV ion beam analytical technique is based on simple elastic scattering of charged particles, Rutherford Backscattering Spectrometry (RBS). RBS is best suited for analyzing high Z elements in a low Z target. It is, however, rather ineffective for cases involving low Z elements in a high Z matrix due to combined effects of a small scattering cross-sections and a high background noise arising from the matrix. For extremely low Z elements, backscattering cannot be used at all since the incident ion (usually He) cannot be scattered beyond 90° by elements lighter than itself.

Because of this limitations in standard RBS, two extensions have been used for analyzing samples containing low Z elements; nuclear reaction analysis (NRA) and forward recoil elastic scattering (FRES). There are serious drawbacks to first including (1) a limited number of applicable reactions, (2) low cross-sections for most reactions, (3) the difficulty of analysis posed by the non-Rutherford cross-sections and (4) need for extremely high energy beams for all but the lowest Z reactions. Consequently, NRA is generally used only in limited cases where no other technique can be applied. The techniques based upon FRES, in contrast, are relatively simple and have been broadly used in numerous materials systems. Rather than observing the scattering of the incident

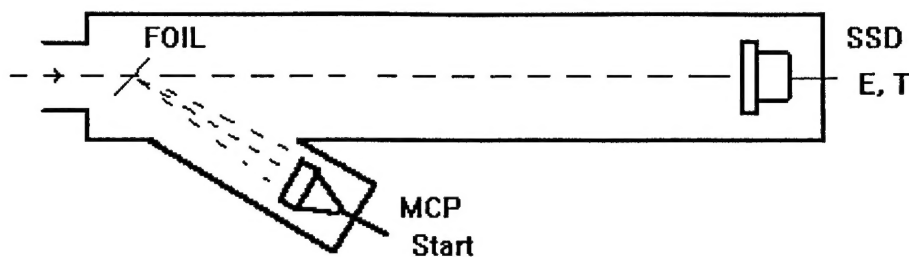


Figure 1: Principal diagram of a Time-of-Flight Energy spectrometer.

ions, FRES analysis depends on observing the recoil of target atoms at angles near 0° . Advantages of FRES include:

- FRES can be easily adopted to use on existing ion accelerators
- scattering physics is relatively simple and standardless quantification of the results is usually possible

However, FRES has several inherent problems. Because the recoiled particles can only be detected in forward scattered directions, both recoiled target ions and scattered incident ions are incident on the detector. The signature from the low Z elements must be extracted from the overall energy spectrum; the number of scattered incident ions though is many orders of magnitude larger than the number of recoiled target ions. Consequently, meaningful data can only be obtained if scattered ions are “filtered out” before reaching the detector. This is usually achieved by placing a thin foil (absorber or stopper) between the sample and detector – sufficiently thick that the incident ions are stopped but thin enough to transmit (with some loss of energy) the recoiled ions. (Because stopping power scales with Z , use of a foil effectively limits FRES to detecting only particles with a lower Z than the incident beam.) This passive filtering method is widely used because of the simplicity and cost. Foils, however, seriously degrade FRES analysis in three ways. First, recoiled ions traversing the foil are broadened in energy by energy straggling leading to dramatically reduced depth resolution. Second, the overall depth of analysis is reduced since recoiled atoms lose energy in the foil and hence low energy recoils cannot be detected. Third, even with an absorber foil, the resulting energy spectrum is a convolution of all particles which can penetrate the foil, complicating the analysis.

3 Variable Geometry ToF Spectrometer System

The limitations of traditional FRES can be overcome only by eliminating the absorber foil completely, and replacing a simple energy sensitive detector with one which (a) differentiates not only by energy but also by ion species, and (b) does not degrade the energy of the ions prior to detection. A detector system based on both energy sensitive surface barrier detectors combined with a Time-of-Flight (ToF) spectrometer satisfies these requirements. The basic principles of such a ToF-E detector system are shown in Figure 1.

As recoiled and scattered particles enter the ToF detector, they first pass through a very thin carbon foil generating a spray of secondary electrons. These secondary electrons are collected, amplified and detected by a micro-channel plate (MCP) which provides the start pulse for a nano-second resolution timing. The ions continue along the 0.98 m path to an energy sensitive surface barrier detector in the flight tube. The current pulse from this detector is split by the preamplifier providing a stop signal to the timer (time of flight) and an input to a conventional multi-channel energy analyzer. For an energy E in MeV and a particle mass m in AMU, the flight time through

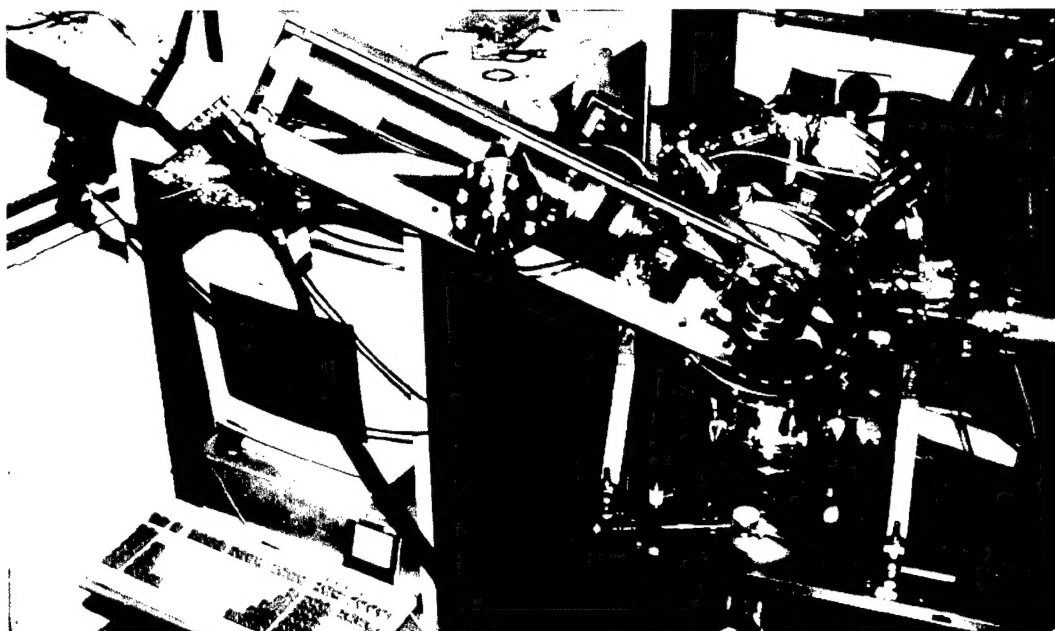


Figure 2: Frontal view of the ToF System. The ToF detector is rotated to 150° position.

the tube is simply

$$\tau = 72.0 L \sqrt{\frac{m}{E}} \quad (1)$$

where τ is in ns and L is in meters. For 1 MeV He ion, it requires approximately 141 ns to travel down the flight length of 0.98 m; nanosecond resolution is thus easily adequate to mass separate the particles.

The ToF detector is attached to the scattering chamber on a unique flange which allows the detector to be rotated to any scattering/recoil angle.

A miniature titanium pump at ToF detector is used to maintain high vacuum while the detector gate valve (located between the detector and the chamber) is closed for sample changing. The chamber itself is evacuated with a powerful 500 l/s turbomolecular pump giving a low 10^{-7} Torr base vacuum in the chamber-detector assembly. The sample holder assembly is a 3x1.5 "paddle" attached to a magnetically coupled load-arm; the load-arm can be moved in and out (z-direction) for sample horizontal positioning and (un)loading. In addition to the z motion, the rod can be rotated around axis for sample tilting (enhanced surface sensitivity). The sample holder assembly itself is easily attached and removed from the load-arm rod and can be electrically isolated for charge integration measurements. A miniature tilt sensor, connected to a meter outside the chamber, provides reliable and accurate measurement of the sample tilt up to a rotation angle of 60°. Clips to secure samples are provided on both sides of the sample holder, though one side usually has a thin quartz slide mounted as a convenient means of visual identifying and aligning the incident ion beam.

The chamber-detector assembly as seen from the sample loading window is shown in figure 2, and from the load arm side in figure 3.

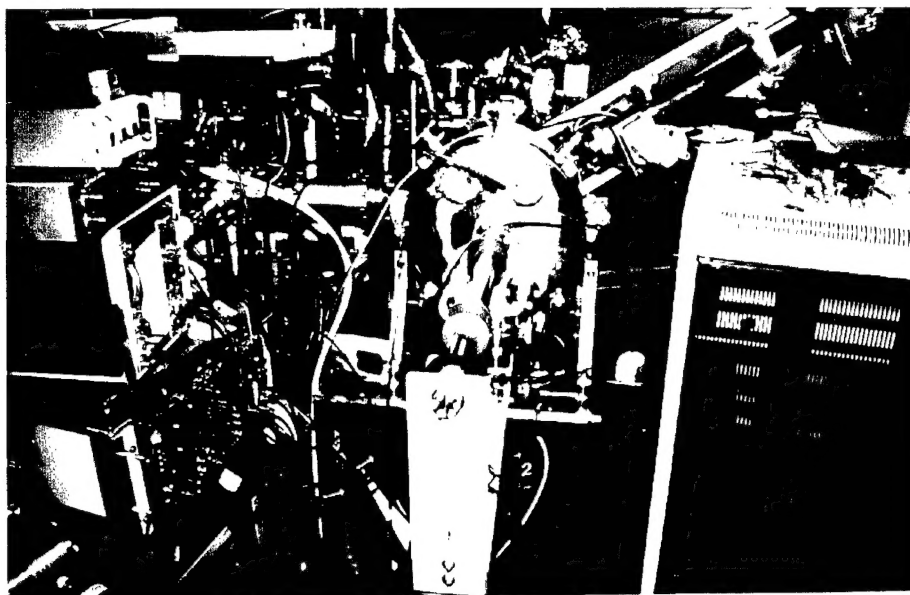


Figure 3: Rear view of the ToF System.

4 Electronics and Spectrum Acquisition

The principle of operation of the electronics setup is relatively simple, as diagrammed in figure 4. The solid-state detector (SSD) at the end of the flight tube is connected to a preamplifier which generates two pulses for each incident ion. The Energy proportional signal is coupled through a spectroscopic amplifier and fed to the first analog-to-digital converter (ADC1). The signal from the Time output of the preamplifier, indicating the arrival of the particle at the SSD, is shaped by a constant fraction discriminator (CFD). Contrary to the simplified description above, this signal is connected to the start signal of the Time-to-Amplitude Converter (TAC), for reasons explained below. Secondary electrons generated by passage of ions through the thin carbon foil are captured and amplified by the micro-channel plate (MCP). The pulse from the MCP is further amplified and shaped with a pico second timing discriminator. It then undergoes a fixed time delay before being fed to the Stop input of the TAC. The TAC then generates an output signal proportional to the time difference between the Start and Stop inputs.

Although, in reality, the ion is first registered by the MCP and later by the SSD, the SSD is used as the start signal and the MCP is the stop signal after being physically delayed. The advantage of this inverse timing solution is obvious: for purely geometrical reasons, the counting rate of the MCP is about one order of magnitude greater than that of the SSD (much larger solid area). Consequently, most of the events from the MCP are false pulses originating from particles that never reach the SSD. With the inverse timing, these events are never registered by the TAC and thus do not contribute to dead time or noise.

The output of the TAC is connected to a second analog-to-digital converter (ADC2) providing a signal proportional to the flight time (ToF). The two ADCs thus provide both the energy and time inputs for a computer-based multi-dimensional data acquisition system (MDA). With the MDA, only time and energy pulses arriving simultaneously (within a defined coincidence window) are registered. The resulting two-dimensional spectrum (ToF vs. Energy) consists of points, each

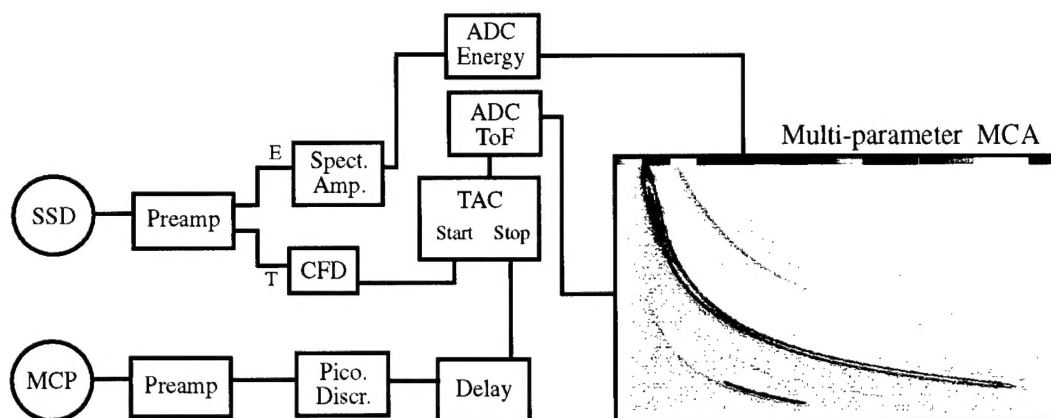


Figure 4: Layout of Electronics of the ToF System.

The commercially available MDA system was delivered with a Windows-based software acquisition package but no data analysis capabilities. The software provided was extremely inefficient and typical 2D spectra required upwards of 2-3 MB per spectrum. In parallel with the hardware deployment, we developed a GUI based OS/2 program, Time of Flight Utility (TOFU), that:

- compresses 2D spectra by 3-10 times for storage efficiency and faster file handling
- selects mass bands and converts them into single ion RBS-like spectra which can be traditionally analyzed
- converts the time domain to an energy domain
- provides for calibration of the 2D spectra in energy, ToF and mass
- provides a convenient interface to the RUMP/Genplot program families for analysis of the resulting 1D spectra

5 Performance Testing ToF Spectrometer System

In figure 5, the 2D ToF spectrum (events in time versus energy space) is shown for a layer of partially deuterated d-TMPC on polystyrene (PS). The horizontal axis represents the particle energy as measured by the SSD with vertical axis giving the time-of-flight. The 2D plot for this sample shows four distinct bands with the meaning of each relatively easy to identify. A vertical line in the 2D plot corresponds to a fixed energy. Particles of different masses with the same energy will be detected with different ToF – the heavier the particle, the longer the ToF. Therefore, events corresponding to particles with different masses occupy separate bands on the 2D representation with the shape of the bands determined by the ToF vs. E dependence, $\sim 1/\sqrt{E}$. The separation of masses into bands in the 2D spectrum gives the greatest advantage of this technique over the conventional absorber foil based FRES. In 2D analysis, contributions of different masses can easily be separated without spectrum overlap. Moreover, the absence of the absorber foil improves the depth resolution and accessible depth. Events in the 2D spectrum that correspond only to recoiled deuterium ions are shown inside the red polygon. These events were extracted and are displayed as a traditional counts versus energy histogram as shown by the dotted black line overlaid on figure 5.

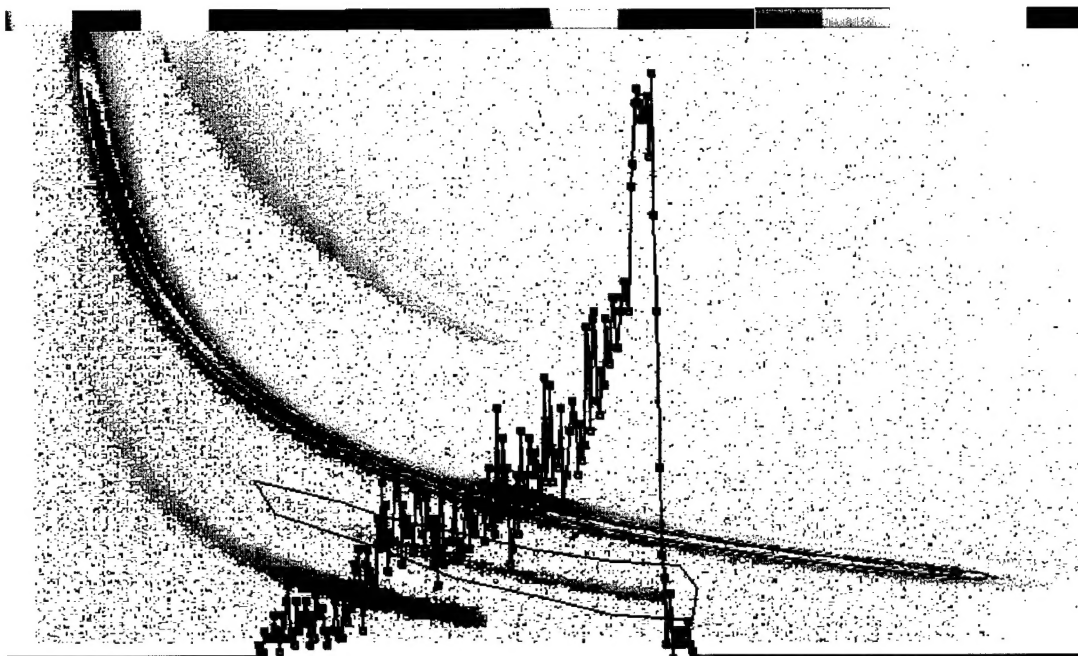


Figure 5: 2D ToF spectrum of a partially deuterated polymer (d-TMPC) layer on PS.

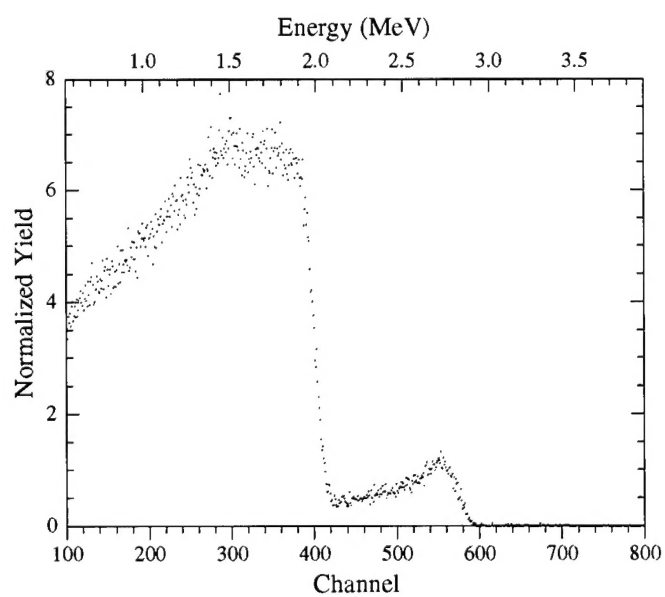


Figure 6: Traditional FRES spectrum of a partially deuterated polymer (d-TMPC) layer on PS

basic feature differences between the two deuterium profiles (see figures 5 and figure 6). First, as a result of dramatically improved depth resolution, a sharp near-surface accumulation of deuterium is observed in the TMPC layer in the ToF spectrum; this feature is almost totally washed out by straggling in the stopper foil. Second, in the conventional FRES spectrum, the tail region of the deuterium profile is lost as the energy begins to overlap with surface scattering of the hydrogen peak. The spectrum obtained from the 2D analysis, on the other hand, reveals the tail region well into the sample.

6 Modification of Tasks

The ion etching system initially proposed was not included in the final hardware for several reasons: Adequate etch rates were not possible to achieve under high vacuum conditions over the areas required for this technique. Funds not expended on the etcher were incorporated into upgrades of the vacuum chamber and the TOF detector/electronics to enhance data acquisition capabilities.

7 Summary

8 Equipment Purchases

| | | |
|------------------------------|---------------------------|------------|
| Sputter Ion Source | Peabody Scientific | 18,000 |
| Custom Chamber | Balzers | 24,200 |
| Vacuum Pumps/Gauges | Balzers | 25,200 |
| Miscellaneous Vacuum | Balzers, Kurt Lesker, MKS | 7,900 |
| Computer System | CSI, HP, Gateway | 5,900 |
| TOF Detector | Xenotek | 36,300 |
| Detector Electronics | Ortec, Fast Comtek | 37,400 |
| in-situ Camera | Electrim Corp | 900 |
| Machine Shop Charges | Cornell Shops | 3,500 |
| | | ===== |
| Total Capital | | \$150,700 |
| SSD Detectors | EG&G Ortec | 1,200 |
| Software/Books | Indelible Blue, MRS | 800 |
| | | ===== |
| Total Expendables | | \$ 2,000 |
| | | |
| Salary (P. Revesz, 25% time) | | 24,349 |
| | | ===== |
| Total Salary/Overhead | | \$ 24,349 |
| | | |
| | | ===== |
| Total Expenses | | \$186,649 |
| | | |
| ARO Contribution | | \$ 150,449 |
| Cornell Contribution | | \$ 36,200 |